

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2008-150627

(43)Date of publication of application : 03.07.2008

(51)Int.Cl.

COB.L 101/10 (2006. 01)
COB.L 71/02 (2006. 01)
COB.K 5/098 (2006. 01)
COB.K 5/17 (2006. 01)
COB.L 23/26 (2006. 01)

(21)Application number : 2008-068520

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(22)Date of filing : 17.03.2008

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(30)Priority

Priority number : 2001226971	Priority date : 27.07.2001	Priority country : JP
2001295770	27.09.2001	JP
2001340999	06.11.2001	JP

(54) CURABLE COMPOSITION

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a curable composition giving a cured product which ensures practical curability and recovery properties and exhibits mechanical properties of high strength and high elongation.

SOLUTION: The curable composition is characterized by comprising (A) an organic polymer having at least one silicon-containing group which has a hydroxy or hydrolyzable group bonded to the silicon atom and is crosslinkable by forming a siloxane bond, and (B) one or more metal salts of a carboxylic acid selected from calcium carboxylate, vanadium carboxylate, iron carboxylate, titanium carboxylate, potassium carboxylate, barium carboxylate, manganese carboxylate, nickel carboxylate, cobalt carboxylate, and zirconium carboxylate.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[Field of the Invention]

[0001]

In this invention, it has the hydroxyl group or hydrolytic basis combined with the silicon atom, and a siloxane bond is formed. Therefore, it is related with the hardenability constituent containing the organic polymer which has a silicon content group (hereinafter a "reactive silicon group") which can construct a bridge.

[Background of the Invention]

[0002]

It is known that the organic polymer which contains at least one reactive silicon group in a molecule has the following character in which construct a bridge by formation of the siloxane bond accompanied by the hydrolysis reaction of a reactive silicon group, etc., and a rubber-like hardened material is obtained with hygroscopic surface moisture etc. also in a room temperature.

[0003]

In the polymer which it has, these reactive silicon groups a polyoxysilane series polymer and a polysiloxane series polymer. It is indicated by JP-S52-73988A, JP-H5-125722A, JP-H3-72527A, JP-S63-6003A, JP-S63-6041A, JP-H1-38407A, JP-H6-231756A, etc.

Especially a polyoxysilane series polymer and a polysiloxane system polymer are already produced industrially, and are widely used for uses, such as a sealing material, adhesives, and a paint.

[0004]

When an organic polymer is a saturated hydrocarbon system polymer especially, since it excels in heat resistance, a water resisting property, weatherability, etc., if the hardened material obtained is used for a structural sealing material, the sealing material for multiple glass, etc., it is effective. The isobutylene system polymer which has a repeating unit which furthermore originates in isobutylene 80% of the weight or more in a total amount is preferred as a sealant aiming its moisture proof from especially the thing to excel in low moisture permeability and low gas permeability.

[0005]

Such a sealing material generally fills up the joined part and cures between various members, the effectiveness to use part ranging from being used to a long period of time in order to get the desired effect. Therefore, it is necessary to obtain a material having sufficient elongation and intensity is called for.

[0006]

Amine compounds, such as lauryl amine, are used together as a co-catalyst in many cases for the purpose of a silanol condensation catalyst being used for bridge construction hardening of an organic polymer which, on the other hand, has such a reactive silicon group, and also accelerating a condensation reaction. As such a silanol condensation catalyst, divalent tin, such as octylic acid (2-ethylhexanoic acid), the tin series catalyst represented by trivalent tin, such as dibutyltin bisacrylate/lactonate, is used widely, and when the movement of the part which pieces especially a sealing material is large, since the hardened material which has stability is obtained, many divalent tin is used.

[0007]

However, when octylic acid tin which is divalent tin, for example is used together with the amine compound of a co-catalyst and is used as a curing catalyst of the (A) ingredient, the intensity and elongation of a hardened material which are acquired may be insufficient, and an improvement of the further physical properties is desired.

[0008]

Although most is a dibutyltin type, the latter tetravalent tin series catalyst being used now. While the hardened material obtained has emollient, i.e., having the character to follow to the long-term stress change from the outside and fast curability, it is broadly used from the ability to apply as a curing catalyst of a liquid mold-curing nature constituent. However, since toxicity in case a small amount of the material contains in this is regarded as questionable, development of the silanol condensation catalyst of a non-tin series is desired.

[0009]

In JP-S35-2765B, JP-S32-3429B, JP-S35-9639B, JP-S37-3271B, etc. As a curing catalyst of an organopolysiloxane constituent, the carboxylic acid metal salt of various kinds of non-tin is indicated with carboxylic acid tin. As an example in which the carboxylic acid metal salt of non-tin is used for the curing catalyst of the constituent which, on the other hand, contains the organic polymer which has a reactive silicon group, although there were carboxylic acid lithium (JP-H5-39428A, JP-H6-12860A) and carboxylic acid cerium (JP-2000-313814A), there was no example broadly examined in various carboxylic acid metal salt until now.

[Patent documents 1] JP-S52-73988A

[Patent documents 2] JP-H5-125722A

[Patent documents 3] JP-H3-72527A

[Patent documents 4] JP-S63-6003A

[Patent documents 5] JP-S63-6041A

[Patent documents 6] JP-H1-38407A

[Patent documents 7] JP-H6-231756A

[Patent documents 8] JP-S35-2765B

[Patent documents 9] JP-S35-9639B

[Patent documents 10] JP-S37-3271B, etc.

[Patent documents 11] JP-S32-3429B

[Patent documents 12] JP-H5-39428A

[Patent documents 13] JP-H6-12860A

[Patent documents 14] JP-2000-313814A

[Description of the Invention]

[Problem(s) to be Solved by the Invention]

[0010]

The purpose of this invention is as follows.

Have practical hardenability and stability with a good hardened material obtained.

Provide a hardenability constituent useful as the structural sealing material asked especially for high durability, the sealing material for multiple glass, the moisture seal material used for an electric electronic component, adhesives, etc. from revealing the outstanding intensity and elongation.

[Means for Solving the Problem]

[0011]

In order that this invention persons may solve such a problem, as a result of inquiring, as a curing catalyst of this (A) ingredient Carboxylic acid calcium, Carboxylic acid vanadium, carboxylic acid iron, carboxylic acid titanium, Carboxylic acid potassium, By using one or more sorts of carboxylic acid metal salt chosen from carboxylic acid sodium, carboxylic acid potassium, carboxylic acid calcium, carboxylic acid vanadium, carboxylic acid iron, carboxylic acid titanium, carboxylic acid potassium, and carboxylic acid calcium, Sealing moderate hardenability and stability, it finds out revealing high intensity and the physical properties of high elongation compared with a case where octylic acid tin is used as a curing catalyst, and came to complete this invention.

[0012]

Namely, this invention has a hydroxyl group or a hydrolytic basis combined with the (A) silicon atom. It is related with a hardenability constituent containing one or more sorts of carboxylic acid metal salt chosen from an organic polymer, (B) carboxylic acid iron, and carboxylic acid cobalt which have at

polymer as oxy metal groups, such as $-ONa$ and $-OK$, first, for example:



Halogen atoms, such as a chlorine atom, a bromine atom, and iodine atoms, and R^1 are among [Type, and Y is $-R^2$, $-R^3-OO-$, or $-R^3-SO-$ (R^2 is a divalent hydrocarbon group of the carbon numbers 1-20, and) an alkylene group, a cyclo alkylene group, an alkylene group, and an aralkylene group — it is desirable — by a divalent organic group shown. — It can obtain by making an organic halogenated compound shown by especially a carbon numbers 1-10 being preferred) react.

[0094]

As a method of using terminal hydroxyl groups of an end hydroxy hydrogenation polybutadiene system polymer as a oxy metal group, A method of making it react to alkaline-water oxides, such as Na, metal alkoxide-NaOH like metal hydride-NaOCH₃ like alkaline metal-NaH like K, and KOH, etc. is raised.

[0095]

Although an end olefin hydrogenation polybutadiene system polymer with the almost same molecular weight as an end hydroxy hydrogenation polybutadiene system polymer used as a starting material is obtained in said method. Before making an organic halogenated compound of a group such as a methylene group, a chloromethyl group, a bromomethyl group, and an iodoalkyl group at the time such as a methylene group, a chloromethyl group, a bromomethyl group, and an iodoalkyl group, it is made to react to an organic halogenated compound which can increase a molecular weight if it is made to react to a multivalent organic halogenated compound which contains two or more halogen in one molecule, and is shown by a general formula (17) after that. A hydrogenation polybutadiene system polymer which is the amount of Polymer Division more, and has an olefin group at the end can be obtained.

[0096]

As an example of an organic halogenated compound shown by said general formula (17), For example, an allyl chloride, an allyl star's picture, vinyl(chloromethyl) benzene, allyl(chloromethyl) benzene, allyl (bromomethyl) benzene, Although allyl (chloromethyl) ether, allyl(chloromethyl) benzene, 1-butenyl (chloromethyl) ether, 1-hexenyl(chloromethyl) benzene, allyloxy (chloromethyl) benzene, etc. are raised. It is not limited to them. Among these, an allyl chloride is cheap, and since it moreover reacts easily, it is desirable.

[0097]

Introduction of a reactive silicon group to said end olefin hydrogenation polybutadiene system polymer may be manufactured by carrying out an addition reaction using a platinum system catalyst in a hydrocarbon compound like a case of an isobutylene system polymer which has a reactive silicon group in molecular chain terminals.

[0098]

When a saturated hydrocarbon system polymer which has a reactive silicon group as mentioned above does not contain substituent, a saturated bond which is not an aromatic ring in a molecule, Compared with a conventional rubber system polymer, which consist of a conventional rubber system polymer like an organic polyisoprene, polybutadiene, and polyisoprene, a vinyl-based system polymer, it is water-resistant because good remarkably. Since this polymer is a hydrocarbon system polymer, its water-resisting property is good, and a low hardened material of humidity permeability is obtained.

[0099]

An organic polymer which has these reactive silicon groups may be used alone, and may be used together two or more sorts. Specifically, a polyoxalkylene series polymer which has a reactive silicon group, a saturated hydrocarbon system polymer which has a reactive silicon group, a vinyl-based polymer which has a reactive silicon group, and an organic polymer which blends two or more sorts chosen from a group, ** and others, can also be used.

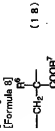
[0100]

A manufacturing method of an organic polymer which blends a polyoxalkylene series polymer which has a reactive silicon group, and a vinyl-based polymer which has a reactive silicon group. Although indicated by JP 69-122651A, JP 63-112662A, JP 68-172651A, JP 63-116763A, etc., it is not limited to in particular these.

[0091]

A desirable example has a reactive silicon group and a chain is a following general formula substantially (18):

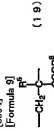
[0092]



[0093]

the acrylic ester monomer unit which has an alkyl group of the carbon numbers 1-8 expressed with (a) hydrogen atom or a methyl group, and R^2 show the alkyl group of the carbon numbers 1-8 among a formula, as for R^2 (meta-), and following general formula (19):

[0094]



[0095]

(— the heads of a formula and R^3 — the same — R^3 shows a with a carbon numbers of ten or more alkyl group —) to the copolymer which consists of an acrylic ester monomer unit which has a with a carbon numbers of ten or more expressed alkyl group (meta-). It is the method of blending and manufacturing the polyoxalkylene series polymer which has a reactive silicon group. Acrylic acid (meta) expresses acrylic acid and/or methacrylic acid with the above-mentioned excessive form.

[0096]

As R^1 of said general formula (18) — the carbon numbers 1-8 of a methyl group, an ethyl group, a propyl group, n-butyl group, t-butyl group, a 2-ethylhexyl group, etc. — desirable — 1-4 — an alkyl group of 1-2 is raised still more preferably. An alkyl group of R^1 may be independent and may be mixed two or more sorts.

[0097]

As R^2 of said general formula (18) — two or more carbon numbers of a lauryl group, a tridecyl group, a eicosyl group, a stearyl group, a behenyl group, etc. — usually — 10-30 — a long-chain alkyl group of 10-20 is raised preferably. Like a case of R^1 , an alkyl group of R^2 may be independent and may be mixed two or more sorts.

[0098]

Although a chain of this vinyl system copolymer consists of a monomeric unit of a formula (18) and a formula (19) substantially, a "real target" here means that the sum total of a monomeric unit of a formula (18) which exists in this copolymer, and a formula (19) surpasses 50 % of the weight. The sum total of a monomeric unit of a formula (18) and a formula (19) is 70 % of the weight or more preferably.

[0099]

From a point of compatibility with a polyoxalkylene series polymer, as for an abundance ratio of a monomeric unit of a formula (18), and a monomeric unit of a formula (19), 95:5-40:60 are preferred at a weight ratio, and 90:10-40:60 are still more preferred.

[0100]

As monomeric units other than a formula (18) which may be contained in this copolymer, and a formula (19), For example, carboxylic acid groups, such as acrylic acid and methacrylic acid, acrylamide, Amide groups, such as methacrylamide, N-methylacrylamide, and N-methylmethacrylamide, Epoxy groups, such as glycidyl acrylate and glycidyl methacrylate, Dimethylamino ethyl acrylate, diethylamino ethyl methacrylate, A monomer containing amino groups.

hardenable constituent of this invention, for example, was described above is blended, it kneads under ordinary temperature or heating using a mixer, a roll, a kneader, etc., or an ingredient is dissolved using a little suitable solvents, and a usual method of mixing may be adopted. A liquid type, two-component type, or many liquid type compound can also be built and used by combining these ingredients suitably.

[0136]

if a hardenability constituent of this invention is exposed into the atmosphere, by operation of moisture, it will form network structure in three dimensions, and will harden it to a solid which has rubber-like elasticity.

[0137]

In a hardenable constituent of this invention, various additive agents can be added if needed. Other curing catalysts as an example of said additive (for example, tetravalent tin, di- or tri-valent tin) are also contemplated.

A adhesive grant agents which adjust the tractive characteristics of a hardened material to generate, such as physical absorbers and a silane coupling agent. An antioxidant radical inhibitor, an ultraviolet ray absorber, a metal deactivator, anti-ozoneant light stabilizer, the Lym system peroxide decomposition agent, lubricants, paints, a foaming agent, a photo-setting resin, a thermoplastic elastomer, etc., may also be used.

The microsilica based agent, alone or mixed

[0138]

Example of such an additive is indicated in each gazette of JP, 4-68659, B, JP, 7-108928, B, JP, 63-254149, A, and JP 64-22904, A, for example.

[0139]

Since a hardenable constituent of this invention has outstanding wettability, heat resistance, water retaining property, and electric insulation resulting from a principal chain skeleton and it has high reactivity and a physical-properties improvement effect of high elongation, it can use as a constituent for electrical insulation materials, such as electrical high elongation component materials, such as structural electric sealing compound, a sealing material for multiple glass, a solar cell near-face sealing agent, pre-impregnation for an electric wire, material for cables, a binder, adhesives, a paint, infusion, a coating material, a sealing agent for pest prevention / water proof, etc. It is useful especially when used as a constituent for structural electric sealing compound, or a sealing material for bonding.

Example

[Example]
[0140] Although working example and a comparative example explain this invention concretely below, this invention is not limited to this.

[0141]

(A) As opposed to isobutylene system polymer (Kaneka Corp. melk. trade name EP005S; isobutylene system polymer / paraffin series process oil = 100/50) 150 weight section which has a reactive silicon group as an ingredient, Epoxy resin (product [made from Oil recovery Shell] Epoxy), trade name Elicostat 828 5 weight section, Hydrogenation alpha-olefin oligomer (product [made from Identical Petrochemistry], trade name PA0504) 80 weight section, Colloid calcium carbonate (marble Cerman Calcium Co. Ltd. melk. trade name SHRETTUSU 20) 50 weight section, Colloid calcium carbonate (Maruo Calcium Co. Ltd. melk. trade name MC-3) 50 weight section, Heavy-calcium carbonate (product [made from Shirotsu Calcium], trade name SOUTON 3200) 40 weight section, Photo-setting resin (Toagosei melk. trade name ARONIKUSSU M-309) 3 weight section, 3921 Benzoinzitol system ultraviolet ray absorbent (Chiba-Gelby Japan melk. trade name SANOPUR LS-770) 1 weight section, Hindered amine light stabiliser (Tokuyama Co. Ltd. melk. trade name ISUCA NOx 1010) 1 weight section, and 10 weight section of the 5 weight section were measured respectively, and it often kneaded the 10 weight section and the 1 weight section, and it was considered as base resin.

[0142]

next — as the (B) ingredient — octylic acid (2-ethylhexanoic acid) calcium salt (the Nihon Kagaku Sangyo Co. Ltd. make). The trade name NIKKAOKU Chinika Ca 5% toluene solution was used as a hardening agent. Lin / for the thing for which KATOYU Chinika (the Kao Corp. make, Firmin D80) is used as a (C) ingredient and which could number / of weight sections / — respectively shown in Table 1 as a (C) ingredient and which could number / of weight sections / — measure, could use the squalene, and was stirred and mixed / working example 1).

[0143]

here = as the (B) ingredient of this invention = naphthalic acid vanadium (the Nihon Kagaku Sangyo Co., Ltd. make), as a trade name NIKKANAFU vinyl 2% toluene solution and the (C) ingredient = distearyl amine (the Kao Corp. make). The number of weight sections = used thing which shows Fmin D88 in Table 1 respectively is made into working example 2, (B) as an ingredient = oxydic acid = used thing which shows respectively laryly amine (made by Wako Pure Chemical Industries, Ltd.) in Table 1 = used thing which shows respectively laryly amine (made by Wako Pure Chemical Industries, Ltd.) in Table 1 as a trade name NIKKAOKU Chikita Fe 6% toluene solution and a (C) ingredient is made into working example 3. The number of weight sections = used thing which furthermore shows respectively laryly amine (made by Wako Pure Chemical Industries, Ltd.) in Table 1 as a (B) ingredient as oxydic acid (2-ethylhexanoic acid) titanium (3% toluene solution) and a (C) ingredient was made into working example 4. On the other hand, the number of weight sections = used thing which shows laryly amine in Table 1 respectively as (B) oxydic acid (2-ethylhexanoic acid) (Japanese east transformation Make, trade name neo SUTAN U-28) and a (C) ingredient was made into the comparative example 1.

[0144]

These base resin and hardening agents were by the ratio shown in Table 1, respectively, and what constrained the aluminum base material to H type in accordance with the manufacturing method of the tensile adhesive property specimen to which this is specified JIS A7578-1992 was followed up. One-of-the health conditions were made into 23 \times 7 \times 1.50 \times 0.5 mm size aluminum used. The aluminum uses the aluminate treatment aluminum of the 50-50 \pm 5 mm size based on JIS H4000 as a substrate. After purifying by methyl ethyl ketone (made by Wako Pure Chemical Industries, Ltd.) before being filled up with the compound, the primer (the Torgy, Indutrade Dow Corning make, trade name: DOWPRIMER) was used under ordinary temperature, for soldering and 30 minutes.

[0145]

In accordance with the tensile cement test method of 5758 - 1992 N of JIS A, the examination was presented with H type sample obtained by the above-mentioned method on condition of speed-of-rotation 50 rpm/min among 23 °C and the thermostatic chamber of 50±5% of humidity.

0034012
[0146]

The recovery was measured by the following methods. First, after mixing H type sample which ended in the above-mentioned case of health immersed into 50 °C warm water for one day and taking it out, it was reflected under the room temperature on the 1st. Subsequently, after having compressed and heated under the room temperature, the sample thickness must be set to 12 to 8.4 mm, and heating a sample in 100 °C fixed to 70% so that sample thickness may be set to 12 to 8.4 mm, and heating a sample in 100 °C hot air drying equipment for one day; it released from compression and was reflected under the room temperature on the 1st. The thickness of the sample in this case was measured and the recovery to a compressed state was computed.

E0147

Time (leather-covered time) until it fills the same compound in an ointment can simultaneously as a measure which estimates hardness and stretches a hide on the surface under 23 ° and the conditions of 50% of relative humidity was measured. The one where leather-covered time is shorter shows that hardness is high.

[0148]

A result is shown in Table 1. The filled hardened material is that cohesive failure is shown, and GF showing the fracture state in front is a fracture state which can be searched for as a sealing material.

[0149]

材料	CO 成分	重量部 (g)									
		1	2	3	4	5	6	7	8	9	10
成分	PPG-3000 3S	50	50	50	50	50	50	50	50	50	50
	7-17700	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
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	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
特性	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
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	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50
	7712300	50	50	50	50	50	50	50	50	50	50

[0150]

Using an isobutylene system polymer as an ingredient (A) as shown in Table 1 as a (B) ingredient, when diisocyanate and lauryl amine were used together for octylic acid calcium, sepiolite acid calcium, and octylic acid titanium as a co-catalyst (working example 1, 2, and 4), the leather-processed material obtained had been secured as compared with the case where octylic acid tin and lauryl amine of the comparative example 1 are used together. On the other hand, in the case (working example 3) where used octylic acid iron as a (B) ingredient, and lauryl amine is used as a catalyst, although it had been 9 hours or more leather-covered time, one day afterward, it checked having hardened enough. The hardened material which was obtained also in the gap to use such carboxylic acid metal salt for (working example 1-4) showed breaking strength with high all, and elongation after fracture for the comparative example 1 which used octylic acid tin and lauryl amine. About stability, although the recovery at the time of using octylic acid titanium and octylic acid iron was lower than the comparative example 1, all had stability. High stability was shown when especially naphthalene acid vanadium was used (working example 2).

[0151]

(Working example 5, comparative example 2)
(A) As a polyether system polymer which has reactant silicon as an ingredient. As opposed to MS polymer S2003 (made by Kinokuni Corp.) 95 weight section, plasticizer (trade name PPG-3000 3S weight section and a dipping inhibitor (made in Kusumoto Chemicals). The product part of trade name DISUPANR 46500 diphens, benzotriazole system ultraviolet ray absorber (Sanyo Co., Ltd. make, trade name THWU-3271) weight section, and hindered amine light stabilizer (Sanyo Co., Ltd. make, trade name SANORU-LS-7700) weight section were added and mixed in excess, the colloid calcium carbonate (trade name Shirakami Industry) which dried this by decompression CCR120 weight section and titanium oxide (Inohara Sangyo Kaisha, Ltd. make, trade name Hakukusa R-820) 20 weight section, and stirring mixing was carried out for 2 hours it mixed to trade name Hakukusa CCR120 weight section, and water content decreased to about 700 ppm with BL planetary mixer again. After it kneaded ones with 3 paint roll, and also checked that dried by decompression stirring at 120 °C for 2 hours, and water content decreased to about 700 ppm with BL planetary mixer again. After cooling a mixture, it added and stirring mixing of the amount part of dehydrator (Nippon Unicar make, trade name A-171) diphens was carried out for 15 minutes. Next, it added and stirring mixing of the aminesilane coupling agent (Nippon Unicar make, trade name A-1120) 3 weight section was carried out for 15 minutes. The octylic acid zirconium (the Nihon Kagaku Sangyo Co., Ltd. make, trade name NIKKAOKU Chicks Zn:2% toluene solution) which is the (B) ingredient adjusted (further beforehand, (C) The number of weight sections which shows lauryl amine (made by Wako Pure Chem) which is an ingredient in Table 2, and the mixed thing were added, and decompression stirring mixing was

performed for 5 minutes. The cartridge made of paper (product made from the Showa round cylinder) by which the coat was carried out was filled up with the aluminum for 1 liquid so that the obtained mixture might not involve in a bubble, and 1 liquid mold-curing nature constituent was obtained. What used the amount part of dibutyltin diacetyl isocyanate (Japanese east transformation Make, trade name U-220) diphens was made into the comparative example 2 instead of the mixture of said octylic acid zirconium and lauryl amine.

[0152]

After replacing the above-mentioned 1 liquid mold-curing nature constituent under a room temperature for 24 hours or more, the speed of the material was added to the mold. What the material was added to H type in accordance with the manufacturing method of the leather adhesive property specimen specified to JIS A5758-1992 using the cancer of marking of 1 liquid mold-curing nature constituent from 1 liquid cartridge was filled up. Care-of health conditions were made into 23 °C, 14 ± 50 %RH, 14 days. [per day] Using the alumite treatment aluminum of the 50:50:50 size based on JIS H4000, before the aluminum used as a substrate was filled up with the compound, it was purified by methyl ethyl ketone (made by Wako Pure Chemical Industries, Ltd.). In accordance with the tensile cement test method of 5758-1992 N of JIS A, the examination was presented with H type sample obtained by the above-mentioned method on condition of 23 °C, and humidity 50 inside of ±5% of thermostatic chamber and speed-of-testing [of 50 mm /]. The hardened material set stress when it elongated 150% to M50, M100, and M150 100%, 50%, respectively, and set TB and elongation at the time of a fracture to EB for the stress at the time of a fracture.

[0153]

Since an adhesive property turned into the important characteristic as a 1 liquid mold-curing nature constituent, the adhesive property was checked using acidified aluminum and a vinyl chloride steel plate. On each substrate which purified the surface by methyl ethyl ketone, 1 liquid mold-curing nature constituent was placed from 1 liquid cartridge using commercial container (made by Wako Pure Chemical Industries, Ltd.) x length of about 3 cm, and it was reconstituted for 14 ± 50 %RH, 14 days per day. The manual traction test was carried out by pulling the adhesive side deeply with a knife, and the fracture surface of the substrate was observed. The filled hardened material is that a fracture failure is shown, and OF in Table 2 is a fracture state searched for as a sealing material.

[0154]

Depth hardness took out the portion which has hardened the surface which filled in the 80-mm-long polyethylene tube 1 liquid mold-curing nature constituent produced in working example 5, and was made at the tip of a tube in it at 16.5 mm in inside diameter on the day which the days to measure vibed, and measured the hardened thickness with slide calipers.

[0155]

In order to check the practicality as a 1 liquid mold-curing nature constituent, where 1 liquid cartridge is filled up with a constituent, it stored for two weeks in the hot air drying equipment which carried out the temperature control to 50 °C, and the depths hardness after storage was investigated. A result is shown in Table 2.

[0156]

[Table 2]

X	A	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ	AK	AL	AM	AN	AO	AP	AQ	AR	AS	AT	AU	AV	AW	AX	AY	AZ	BA	BB	BC	BD	BE	BF	BG	BH	BI	BJ	BK	BL	BM	BN	BO	BP	BQ	BR	BS	BT	BU	BV	BW	BX	BY	BZ	CA	CB	CC	CD	CE	CF	CG	CH	CI	CJ	CK	CL	CM	CN	CO	CP	CQ	CR	CS	CT	CU	CV	CW	CX	CY	CZ	DA	DB	DC	DD	DE	DF	DG	DH	DI	DJ	DK	DL	DM	DN	DO	DP	DQ	DR	DS	DT	DU	DV	DW	DX	DY	DZ	EA	EB	EC	ED	EE	EF	EG	EH	EI	EJ	EK	EL	EM	EN	EO	EP	EQ	ER	ES	ET	EU	EV	EW	EX	EY	EZ	FA	FB	FC	FD	FE	FF	FG	FH	FI	FJ	FK	FL	FM	FN	FO	FP	FQ	FR	FS	FT	FU	FV	FW	FX	FY	FZ	GA	GB	GC	GD	GE	GF	GG	GH	GI	GJ	GK	GL	GM	GN	GO	GP	GQ	GR	GS	GT	GU	GV	GW	GX	GY	GZ	HA	HB	HC	HD	HE	HF	HG	HH	HI	HJ	HK	HL	HM	HN	HO	HP	HQ	HR	HS	HT	HU	HV	HW	HX	HY	HZ	IA	IB	IC	ID	IE	IF	IG	IH	II	IJ	IK	IL	IM	IN	IO	IP	IQ	IR	IS	IT	IU	IV	IW	IX	IY	IZ	JA	JB	JC	JD	JE	JF	JG	JH	JI	JJ	JK	JL	JM	JN	JO	JP	JQ	JR	JS	JT	JU	JV	JW	JX	JY	JZ	KA	KB	KC	KD	KE	KF	KG	KH	KI	KJ	KK	KL	KM	KN	KO	KP	KQ	KR	KS	KT	KU	KV	KW	KX	KY	KZ	LA	LB	LC	LD	LE	LF	LG	LH	LI	LJ	LK	LM	LN	LO	LP	LQ	LR	LS	LT	LU	LV	LW	LX	LY	LZ	MA	MB	MC	MD	ME	MF	MG	MH	MI	MJ	MK	ML	MM	MN	MO	MP	MQ	MR	MS	MT	MU	MV	MW	MX	MY	MZ	NA	NB	NC	ND	NE	NF	NG	NH	NI	NJ	NK	NL	NM	NN	NO	NP	NQ	NR	NS	NT	NU	NV	NW	NX	NY	NZ	OA	OB	OC	OD	OE	OF	OG	OH	OI	OJ	OK	OL	OM	ON	OO	OP	OQ	OR	OS	OT	OU	OV	OW	OX	OY	OZ	PA	PB	PC	PD	PE	PF	PG	PH	PI	PJ	PK	PL	PM	PN	PO	PP	PQ	PR	PS	PT	PU	PV	PW	PX	PY	PZ	QA	QB	QC	QD	QE	QF	QG	QH	QI	QJ	QK	QL	QM	QN	QO	QP	QQ	QR	QS	QT	QU	QV	QW	QX	QY	QZ	RA	RB	RC	RD	RE	RF	RG	RH	RI	RJ	RK	RL	RM	RN	RO	RP	RQ	RR	RS	RT	RU	RV	RW	RX	RY	RZ	SA	SB	SC	SD	SE	SF	SG	SH	SI	SJ	SK	SL	SM	SN	SO	SP	SQ	SR	SS	ST	SU	SV	SW	SX	SY	SZ	TA	TB	TC	TD	TE	TF	TG	TH	TI	TJ	TK	TL	TM	TN	TO	TP	TQ	TR	TS	TT	TU	TV	TW	TX	TY	TZ	UA	UB	UC	UD	UE	UF	UG	UH	UI	UJ	UK	UL	UM	UN	UO	UP	UQ	UR	US	UT	UU	UV	UW	UX	UY	UZ	VA	VB	VC	VD	VE	VF	VG	VH	VI	VJ	VK	VL	VM	VN	VO	VP	VQ	VR	VS	VT	VU	VV	VW	VX	VY	VZ	WA	WB	WC	WD	WE	WF	WG	WH	WI	WJ	WK	WL	WM	WN	WO	WP	WQ	WR	WS	WT	WU	WV	WW	WX	WY	WZ	XA	XB	XC	XD	XE	XF	YG	YH	YI	YJ	YK	YL	YM	YN	YO	YP	YQ	YR	YS	YT	YU	YV	YW	YX	YY	YZ	ZA	ZB	ZC	ZD	ZE	ZF	ZG	ZH	ZI	ZJ	ZK	ZL	ZM	ZN	ZO	ZP	ZQ	ZR	ZS	ZT	ZU	ZV	ZW	ZX	ZY	ZZ
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[0127]

When the various carboxylic acid metal salt of working example 16-28 is used as shown in Table 4, in spite of having used the silanol condensation catalyst of the non-tin series, the surface has hardened in five days.

Practical manufacturability was shown. On the other hand, when the carboxylic acid zinc of the comparative example 4 was used, hardnessability had not hardened five days after had

[01 78]

As mentioned above, the organic polymer which has at least one reactive silicon group in the (A) molecule, (B) Carboxylic acid calcium, carboxylic acid vanadium, carboxylic acid iron, Carboxylic acid titanium, Carboxylic acid calcium, carboxylic acid barium, Carboxylic acid manganese, carboxylo acid nickel, carboxylic acid cobalt, one or more sorts of carboxylic acid metal salt chosen from a carboxylic acid zirconium, and a hardener [* and others] constituent, in spite of being a non-tin organotin compound, is abundant, and it became clear that hardened material physical properties are excellent.

.IP 2008-150627 A [DETAILED DESCRIPTION]

properties are also fitness (high elongation and high intensity).

[0179]

Constituent using the trimethoxysilyl group as a reactive silane group)¹ If a trimethoxysilyl group uses the polyoxypolysilane series polymer which exists in molecular chain terminals as a reactive silane group, a cure rate will become larger from the polymer of the synthetic example 1. The division of a trimethoxysilyl group of this is because reactivity is larger than a methyl dimethoxy silane group. Such a polymer is indicated to JP-H11-12480A or JP-2001-72855A. For example, the same hardenable constituent as working example 6-15 can be prepared using the polymer indicated for the working example of JP-H11-12480A. The tack-free time of this constituent is shorter than working example 6-15.

The same hardenability constituent as working example 6-15 can be prepared using the polymer indicated for the example 1-4 of manufacture of JP, 2001-72855A. The tack free time of this constituent is shorter than working example 6-15. Therefore, a catalyst amount can be lessened, if cure time is made into the same grade and the polymer which has the above-mentioned triethoxysilane group will be used.

[0181]

If the mixture of the polymer which has a trimethoxysilyl group, and the polymer which has a methyl dimethoxy silyl group is used as a polymer, cure time and the physical properties of a hardened material are freely controllable. For example, the hardenability constituent of working example 8-15 can be prepared using the polymer which mixed the polymer which has a trimethoxysilyl group, and the polymer which has a methyl dimethoxy silyl group by the weight ratio of 1:10-10:1.

[0192]

Working example when the monomer which has a trimethoxysilyl group is used is shown below.

[0183]

[illegible]

[U184]
(Mordanting example 29-22)

(A) The polyoxazoline series polymer (A-1) which has as an ingredient the methyl dimethoxysilyl group obtained in the synthetic route (1). The various carboxylic acid metal salt which is the (B) ingredient according to the formula shown in Table 5 using the polyoxazoline series polymer (A-3) which has the carboxylic group obtained in the synthetic route (3). (C) Lauryl amine which is an ingredient as measured, respectively, the spatula was used, and it stirred and mixed for 30 seconds. It made for all the numbers of moles of the metal atom containing the number of addition parts. The various carboxylic acid metal salt of the (B) ingredient, to become almost the same here. The surface was lightly pressed down with the spatula after mixing and time (leather-covered time) until a constituent stops adhering at the tip of a spatula was measured. An evaluation result is shown in Table 5.

Table 5.

[0185]
[Table 5]

<http://www.indinfta.in/in/cari-hin/evan-ueh-cm-ess?shw=chttt%3A%2F%2Fwww4.ipdli...>

http://www4.indl.inn.it.go.jp/cgi-bin/tranweb.cgi/ejie?atw_u=http%3A%2F%2Fwww4.ipdl.i... 2010/05/08

組成 (重量部)					
高粘度					
(A) 成分	A-1	29	30	31	32
A-3					
(B) 成分	三好ケイ酸触媒 (特)	100	100	100	100
	日本化学産業 (株)	6.5	6.4		
(C) 成分	三好ケイ酸触媒 (特)			5.8	5.8
	日本化学産業 (株)				
	ラウリルアミン	0.85	0.85		
	4TBA	4TBA	2TBA	10TBA以上	10TBA以上
硬化温度 (24℃)					

(1) : 2-エポキシヘキサンのメタカリスリット溶液、金属 (Co) 含有量 : 5 %

(2) : 2-エポキシヘキサンのメタカリスリット溶液、金属 (Co) 含有量 : 5 %

[0186]

As shown in Table 5, the polyoxalkylene series polymer (A-3, working example 30 and 32) which has a trimethoxysilyl group had the cure rate quicker than the polyoxalkylene series polymer (A-1, working example 29 and 31) which has a methoxy dimethoxy silyl group.

[0187]

The same hardenability constituent as working example 6-15 can be prepared using the polyoxalkylene series polymer (A-3) which has the trimethoxysilyl group obtained in the synthetic method of example 3. The task free time of this constituent has short cure time, and good physical properties are acquired.

[0188]

(Constituent which used the epoxy resin together)

The same hardenability constituent as working example 6-15 can be prepared using the constituent which used together reactive silicon group containing organic polymer and an epoxy resin. When this constituent is used, the manifestation of adhesive strength is quick.

[Industrial applicability]

[0189]

(A) The organic polymer which has the hydroxyl group or hydrolytic basis combined with the silicon atom, and has at least one silicon content group which can construct a bridge by forming a siloxane bond. (B) Carboxylic acid calcium, carboxylic acid vanadium, carboxylic acid iron, Carboxylic acid titanium, carboxylic acid potassium, carboxylic acid barium. The hardenability constituent containing one or more sorts of carboxylic acid metal salt which it comes to choose out of carboxylic acid manganese, carboxylic acid nickel, carboxylic acid cobalt, and a carboxylic acid zirconium. While revealing practical hardenability and stability, the hardened material obtained shows the outstanding mechanical property which has high intensity and high elongation as compared with the case where the carboxylic acid tin currently used conventionally is used as curing catalyst. Therefore, the hardenability constituent of this invention is very useful as various elastomers, such as a sealing material, adhesives, a binder.

[Translation done.]